IS 6301 : 2013
(Reaffirmed 2019)

भारतीय मानक

क्षारीय क्रोमियम सल्फेट (चर्म शोधन के लिए) — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

BASIC CHROMIUM SULPHATE (FOR TANNING) — SPECIFICATION

(First Revision)

ICS 59.140.10

© BIS 2013

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

August 2013 **Price Group 5**

Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Leather, Tanning Materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

Basic chromium sulphate is used in leather industry mainly for the chrome tanning process of leather manufacture. This chrome salt may be prepared, adjusted to various basicities to suit the need of individual tanner. But in this standard, the percentage basicity of the salt which finds wide application in tanning process has only been prescribed. Characteristics like chromium content, percentage basicity, free sulphate, moisture, *p*H of aqueous extract and matter insoluble in water have been prescribed.

This standard was originally published in 1971. In this revision, the following changes have been made:

a) Requirement on pentachloro phenol (PCP) has been introduced keeping in view of the demand for eco-friendly inputs from the leather industry.
b) Requirement of unreduced chromium has been replaced by the requirement of hexavalent chromium.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BASIC CHROMIUM SULPHATE (FOR TANNING) — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for basic chromium sulphate, in the form of flakes or powder, used in leather industry for tanning.

2 REFERENCES

The following standards are necessary adjuncts to this standard:

IS No.	*Title*
264 : 2005	Specification for nitric acid (*third revision*)
265 : 1998	Specification for hydrochloric acid (*fourth revision*)
266 : 1993	Specification for sulphuric acid (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
14575 :1999	Determination of pentachloro phenol (PCP) in leather — Method of test

3 REQUIREMENTS

3.1 Description

Basic chromium sulphate shall be in the form of flakes or powder.

3.1.1 A 5 percent aqueous solution of basic chromium sulphate shall be transparent and optically clear.

NOTE — A solution is to be considered as optically clear if a bright object is clearly visible through 5 cm thick layer and if a 1 cm layer in a beaker placed on black glass or a paper appears black and not opalescent when viewed from above in a good light.

3.2 Composition

The material shall comply with the requirements given in Table 1, when tested according to the methods prescribed in Annex A. Reference to relevant clauses of Annex A is given against each of the characteristics in col 4 of Table 1.

4 PACKING

The material shall be packed as agreed to between the purchaser and the supplier.

5 MARKING

5.1 The packages shall be securely closed and marked with the following information:

a) Name of the material;
b) Net weight of the material in the package;
c) Year and month of manufacture and the lot number in code or otherwise to enable the batch of manufacture to be traced back from records;
d) Type of reducing agents used (organic or inorganic); and
e) Manufacturer's name and/or trade-mark, if any.

5.2 BIS Certification Marking

The packages may also be marked with the Standard Mark.

Table 1 Requirements for Basic Chromium Sulphate (for Tanning)
(*Clause* 3.2)

Sl No. (1)	Characteristic (2)	Requirements (3)	Method of Test, Ref to (4)
i)	Moisture, percent by mass, *Max*	10.0	A-2
ii)	Free sulphate (as Na_2SO_4), percent by mass, *Max*	35.0	A-3
iii)	Basicity, percent	30-35	A-4
iv)	Chromium (as Cr_2O_3), percent by mass, *Min*	24	A-4
v)	Matter insoluble in water, percent by mass, *Max*	0.2	A-5
vi)	*p*H of aqueous solution	3.0 ± 0.2	A-6
vii)	Hexavalent chromium, mg/kg, *Max*	3	A-7
viii)	PCP content, mg/kg	5	A-8

5.2.1 The use of the Standard Mark is governed by the provision of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING AND CRITERIA FOR CONFORMITY

The method of drawing representative samples of the material, number of test to be performed and criteria for conformity of the material to the requirements of this standard shall be as prescribed in Annex B.

ANNEX A

(*Clause* 3.2)

METHOD OF TEST FOR BASIC CHROMIUM SULPHATE (FOR TANNING)

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF MOISTURE

A-2.1 Apparatus

A-2.1.1 *Air Oven*, with thermostatic control capable of maintaining at 105 ± 2°C.

A-2.1.2 *Wide Mouth Weighing Bottle*

A-2.2 Procedure

Transfer about 1 g of basic chromium sulphate into a tared wide mouth weighing bottle and weigh accurately. Dry at about 105 ± 2°C in an air oven for 3 to 4 h. Cool in a desiccator for about 20 min and weigh again accurately. Repeat the process of drying and weighing until two weightings at an interval of 1 h do not differ by more than 2 mg.

NOTE — If the weight of the dried material is found to increase on longer drying, then the lowest weight found shall be taken.

A-2.3 Calculation

$$\text{Moisture, percent by mass} = \frac{(W_1 - W_2) \times 100}{W_1}$$

where

W_1 = mass of the material taken for the test, in g; and

W_2 = mass of the material after drying, in g.

A-3 DETERMINATION OF FREE SULPHATE (AS Na_2SO_4)

A-3.1 Outline of the Method

The total sulphate in terms of Na_2SO_4 and the sulphate combined with chromium is determined. From the difference of these two, the percentage of free sulphate is calculated.

A-3.2 Reagent

A-3.2.1 *Barium Chloride Solution*, approximately 10 percent.

A-3.2.2 *Dilute Hydrochloric Acid*, 1.5 N.

A-3.2.3 *Standard Sodium Hydroxide Solution*, 0.5 N or 0.2 N.

A-3.2.4 *Phenolphthalein Solution*, 1 percent alcoholic, neutralized solution.

A-3.3 Procedure

A-3.3.1 *Determination of Sulphate Combined with Chromium*

Weigh accurately a suitable quantity of basic chromium sulphate and dilute to 250 ml to give a concentration of 1 percent chromium (*see* Note). Pipette out 25 ml of this solution into a large porcelain basin and dilute with approximately 300 ml of water. Add 3 to 4 ml of phenolphthalein solution and titrate, cold with standard sodium hydroxide solution until a pink colour develops. Bring the contents of the basin to boil and with constant stirring continue the titration until the end point is reached. This is shown by greyish violet colour of the well stirred liquor or a pink supernatant liquor colour persists, which indicates that the end point has been achieved.

NOTE — Usually the solution will require about 15 g of solid basic chrome sulphate to have a concentration of 1 percent chromium.

A-3.3.1.1 *Calculation*

The titre of standard alkali consumed corresponds to the acid combined with chromium.

1.0 ml of 0.5 N sodium hydroxide solution = 0.024 g of SO_4 = 0.035 5 g of Na_2SO_4, and

1.0 ml of 0.2 N sodium hydroxide solution = 0.009 6 g of SO_4 = 0.014 2 g of Na_2SO_4.

Express the result as percentage of mass of the original material.

A-3.3.2 *Determination of Total Sulphate*

Filter the residue remaining in the porcelain basin after the titration, carried out in **A-3.3.1** and wash thoroughly. Concentrate the filtrate and washings to 250 ml by evaporation on a steam-bath. Pipette 100 ml of aliquot of the solution into a 400-ml beaker, provided with a stirring rod and glass cover. Add 10 ml of dilute hydrochloric acid. Heat the solution to boiling. Add dropwise from a burette or pipette 10 to 15 ml of warm barium chloride solution, while stirring the solution. Allow the precipitate to settle for a minute or two. Then test the supernatant liquid for complete precipitation by adding a few drops of barium chloride solution. Repeat this operation of testing for any precipitate in the supernatant liquid until an excess of barium chloride is present. Continue boiling on a steam bath for 15 min and allow it to stand for 4 h. Filter the solution through filter paper (Whatman No. 42 or equivalent). Wash the precipitate with dilute hydrochloric acid and repeatedly with hot water till it is free from chloride. Dry the filter paper and the contents in an air oven, transfer it to a tared silica dish, char the paper over a non-luminous Bunsen flame and heat till all carbon has burnt away, cool, carefully moisten with a few drops of concentrated nitric acid, evaporate off the acid, fume to dryness after moistening with two drops of dilute sulphuric acid and finally muffle it at 600 to 700°C to constant weight.

NOTES

1 Properly prepared Gooch crucible, G No. 4 may also be used for the filtration. In that case the precipitate may be dried in air oven at 110 ± 5°C, cooled and weighed to a constant weight.

2 Excess of barium chloride is necessary to reduce the solubility of barium sulphate.

A-3.3.2.1 *Calculation*

$$\text{Total sulphate (as } Na_2SO_4\text{), percent by mass} = \frac{60.86\, W_1}{W_2}$$

where

W_1 = mass of the ignited precipitate, in g; and

W_2 = mass of the material taken for the test in g.

A-3.3.3 Obtain total sulphate (X) as in **A-3.3.2.1** and sulphate combined with chromium (Y) as **A-3.3.1.1**, then (X–Y) will represent the free sulphate.

A-4 DETERMINATION OF BASICITY

A-4.1 Principle of Method

Basicity of basic chromium sulphate is defined as the percentage of total chrome which is combined with the hydroxyl groups, that is,

$$\text{Basicity, percent} = \frac{(\text{Cr combined with OH} \times 100)}{\text{Total chromium}}$$

It may be calculated as follows:

If V_1 ml of 0.1 N titrant is required in the chromium titration and V_2 ml of 0.1 N sodium hydroxide solution for a similar volume in the acid titration,

$$\text{Basicity, percent} = \frac{(V_1 - V_2)\times 100}{V_1}$$

Therefore, in this method firstly the amount of chromium in the basic chromium sulphate is determined and then the amount of sulphate combined with chromium is determined. From this the percentage basicity is calculated.

A-4.2 Reagents

A-4.2.1 *Sodium Hydroxide Solution* — 1 N.

A-4.2.2 *Hydrogen Peroxide* — 35 percent (*w*/*w*).

A-4.2.3 *Concentrated Hydrochloric Acid* — See IS 265.

A-4.2.4 *Perchloric Acid* — 60 to 70 percent solution.

A-4.2.5 *Standard Sodium Thiosulphate Solution* — 0.1 N freshly standardized.

A-4.2.6 *Standard Ferrous Ammonium Sulphate* — 0.1 N freshly standardized.

A-4.2.7 *Potassium Iodide Solution* — 10 percent (*w*/*v*).

A-4.2.8 *Starch Solution* — Triturate 5 g of starch and 0.01 g of mercuric-iodide with 30 ml of water in a mortar. Pour the resulting paste into one litre of boiling water, boil for 3 min, allow the solution to cool. Decant off the clear liquid.

A-4.2.9 *Nitric Acid* — See IS 264.

A-4.2.10 *Sulphuric Acid* — See IS 266.

A-4.2.11 *Perchloric-Sulphuric Acid Mixture* — Mix 1/3 and 2/5 proportion of perchloric acid and sulphuric acid respectively.

A-4.2.12 *n-Phenyl Anthranilic Acid Solution*

A-4.3 Procedure

A-4.3.1 *Determination of Chromium*

Either of the two methods shall be followed. But, in case of dispute, Method A shall be followed.

A-4.3.1.1 *Method A*

Weigh about 15 g of basic chrome sulphate and dilute to 250 ml to give a concentration of about 1 percent chromium. Pipette out 25 ml of this solution into a conical flask and add sufficient sodium hydroxide solution, dropwise until the precipitate initially formed redissolves. Add 15 to 20 ml hydrogen peroxide, place a funnel or similar devise in the mouth of the flask to prevent loss and bring to the boil, taking care to avoid excessive frothing. Continue boiling to complete the oxidation of the chromium. To ensure complete decomposition of excess peroxide, add a small piece of clean iron and continue boiling until the iron rattles against the glass (generally less than 5 min is required). Filter the solution into a 250 ml graduated flask, wash the precipitate thoroughly with hot water and add the washings to the filtrate. Cool and make upto 250 ml. Pipette out 50 ml of the solution into a stoppered flask, neutralize with hydrochloric acid as indicated by the colour change from yellow chromate to orange dichromate and add 5 ml excess hydrochloric acid. Cool to 27± 2°C, add 10 ml of 10 percent potassium iodide solution, moisten the stopper with the same reagent. Allow the flask to stand for 10 min in the dark. Wash the stopper and the neck of the flask with water into the solution and titrate with 0.1 N sodium thiosulphate solution adding the starch indicator when the titration is almost complete.

NOTE — Any insoluble matter observed in making up the analytical solution should be reported.

A-4.3.1.2 *Method B*

Transfer a measured volume of basic chromium sulphate solution, containing approximately 0.05 g of chromium to a 500 ml conical flask, provided with a funnel in the mouth to act as a trap. Add 20 ml concentrated nitric acid followed by 20 ml of the perchloric-sulphuric acid mixture. Heat the flask and contents gently at the boil until the mixture becomes a pure orange-red colour.

Continue boiling for 1 min after this point has been reached. Remove the flask from the source of heat and as soon as ebullition has ceased; cool rapidly by swirling the flask in a bath of cold water. Carefully add 100 ml water, a few glass beads or anti-bump granules and boil for 10 min to remove free chlorine. Cool and titrate the solution using either sodium thiosulphate solution or ferrous ammonium sulphate solution. If the solution contains iron, the ferrous ammonium sulphate titration procedure shall be used, otherwise follow thiosulphate titration procedure.

A-4.3.1.2.1 *Using sodium thiosulphate solution*

Cool the solution to 27 ± 2°C and transfer to an iodine flask. Add 10 ml of potassium iodide solution, moisten the stopper with the same reagent and replace. Allow the flask to stand for 10 min in the dark. Wash the stopper and the neck of the flask with distilled water into the solution, and titrate with 0.1 N sodium thiosulphate solution, adding the starch indicator when the titration is almost complete.

A-4.3.1.2.2 *Using ferrous ammonium sulphate solution*

Add 10 ml of 30 percent sulphuric acid and cool the flask and contents to 27 ± 2°C. Titrate with freshly standardized 0.1 N ferrous ammonium sulphate solution using six drops of *n*-phenyl anthranilic acid solution as indicator. The end point is indicated by a colour change from violet to green.

A-4.3.1.3 Calculate the chromium content as percentage by mass on the original material using the factor 1 ml of 0.1 N titrant = 0.001 73 g Cr = 0.002 53 g Cr_2O_3.

A-4.3.1.4 *Determination of acid combined with chromium*, same as **A-3.3.1**.

A-4.3.2 *Calculation of Basicity*

If a ml of 0.1 N sodium thiosulphate are required in the chromium titration and b ml of 0.1 N sodium hydroxide for a similar volume in the acid titration, then

$$\text{Basicity, percent} = \frac{(a-b)\times 100}{a}$$

A-5 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-5.1 Apparatus

A-5.1.1 *Steam Bath*

A-5.1.2 *Vacuum Oven*, with thermostatic control capable of maintaining at 100°C.

A-5.2 Procedure

Dissolve 10 g of the basic chrome sulphate in 1 000 ml of water at 27 ± 2°C within a period of 30 min. Filter through a dry filter paper, Whatman No. 42 or equivalent, into a dry beaker. Discarding the first 20-25 ml of filtrate, measure out 250 ml of the filtrate in another beaker or better, in a dish and evaporate on a steam bath. Dry the residue at 100 ± 2°C in a vacuum oven, cool in a desiccator for about 20 min and weigh accurately. Repeat the process of drying and weighing until two weighings at an interval of 1 h do not differ by more than 2 mg.

A-5.3 Calculation

Calculate matter insoluble in water, percent by mass as given below:

$$\text{Matter insoluble in water, percent by mass} = 100 - (40\,W + M)$$

where

W = mass of the residue left after evaporating 250 ml of the solution, in g; and

M = moisture percent by mass.

A-6 DETERMINATION OF *p*H

A-6.1 Apparatus

A-6.1.1 p*H Meter*, with glass electrode.

A-6.2 Procedure

Dissolve about 15 g of basic chrome sulphate in 250 ml water at 27 ± 2°C to give a concentration of about 1 percent chromium. Measure the p*H* of the solution immediately by using a suitable p*H* meter.

NOTE — The p*H* of chrome liquors depend not only on the concentration but on ageing, temperature of preparation or dilution. For results to have any comparative value, the strength of the solution should be the same and should be prepared in an identical temperature and the p*H* should be measured immediately after the preparation of the solution.

A-7 TEST FOR HEXAVALENT CHROMIUM

A-7.1 Apparatus

A-7.1.1 *Volumetric Flasks*, 25 ml, 100 ml.

A-7.1.2 *Pipettes*, nominal volumes 0.5/1.0/2.0/5.0/10 ml.

A-7.1.3 *Spectrophotometer or Filter Photometer*, wavelength 540 nm.

A-7.1.4 *Photometric Cell*, glass/quartz, 2 cm length or any other suitable cell length.

A-7.1.5 *Orbital Shaker*, 50 rev/min.

A-7.2 Reagent

A-7.2.1 *Diphenylcarbazide Solution* — Dissolve 1.0 g of 1,5-diphenylcarbazide [CO $(NHNHC_6H_5)_2$] in 100 ml acetone [(CH_3) CO] and make it acidic with one drop of glacial acetic acid [CH_3COOH]. Store the solution in a brown glass bottle. The shelf-life is up to 14 days at 4°C.

A-7.2.2 *Oxygen-Free Inert Gas* — Argon or nitrogen.

NOTE — Argon is preferred as it has higher specific weight than air.

A-7.2.3 *Phosphoric Acid Solution* — Prepare 70 percent solution by diluting 700 ml *o*-phosphoric acid of density 1.71 g/ml to 1 000 ml with distilled water.

A-7.2.4 *Potassium Dichromate ($K_2Cr_2O_7$) of Analytical Reagent Grade* — Dry the substance for 16 ± 2 h at 102 ± 2°C.

A-7.2.5 *Buffer Solution* — Dissolve 22.8 g dipotassium hydrogen phosphate $K_2HPO_4 \cdot 3\,H_2O$ in 1 000 ml water, and adjust to *p*H 8.0 ± 0.1 with phosphoric acid (*see* **A-7.2.3**).

A-7.2.6 *Chrome VI Stock Solution* — Dissolve 2.829 g potassium dichromate ($K_2Cr_2O_7$) in water in a volumetric flask and make up to 1 000 ml with water.

NOTE — 1 ml of this solution contains 1 mg of chromium.

A-7.2.7 *Chrome VI Standard Solution* — Pipette 1 ml of solution (*see* **A-7.2.6**) into a 1 000 ml volumetric flask and make up to the mark with distilled water.

NOTE — 1 ml of this solution contains 1 µg chromium.

A-7.3 Procedure

A-7.3.0 Determine the amount of hexavalent chromium in basic chromium sulphate by this method after completing the sample preparation in aqueous phosphate buffer solution *p*H at 7.5-8.0.

A-7.3.1 *Extraction of Hexavalent Chromium from Basic Chromium Sulphate*

Weigh 2.0 ± 0.01 g of basic chromium sulphate accurately. Transfer 100 ml of phosphate buffer at *p*H 8.0 (*see* **A-7.2.4**) in a 250 ml flask and displace oxygen available in the solution by passing oxygen-free inert gas (*see* **A-7.2.2**) (50±10 ml/min) for about 5 min into the 250 ml flask. Remove the aeration tube, add the weighed basic chromium sulphate sample to this degassed buffer solution and close the flask with a stopper to air-tight. To effect the extraction, use the orbital shaker (*see* **A-7.1.5**) for 1 h.

Caution — Do not set the shaker at a higher speed for this extraction.

After the extraction, filter the extract through a pleated Whatman No. 42 filter paper or a glass wool filter of 1 micron (or finer) to remove all insoluble particles. The solution after filtration should be clear; if it is not, use an aqueous-compatible membrane filter of around 0.4 microns or employ a centrifuge to clarify the extracts by opting a speed of around 10 000 rpm for 10 min.

A-7.3.2 Pipette 10 ml of the extract solution (*see* **A-7.3.1**) into a 25 ml volumetric flask. Dilute the solution to three-fourth of the volume of flask with buffer solution (*see* **A-7.2.5**). Add 1 ml of diphenylcarbazide solution and afterwards 1 ml of phosphoric acid (*see* **A-7.2.3**). Then make up flask to volume with buffer solution and mix well.

A-7.3.2.1 Allow to stand for 15 ± 5 min. Measure the extinction of the solution at 540 nm in a 2 cm cell against the blank solution (*see* **A-7.5**). Register the extinction obtained as E_1.

A-7.4 Determination of the Self Extinction of the Eluate

Pipette out another 10 ml aliquot of the extract solution obtained in **A-7.3.1** into a 25 ml volumetric flask and treat it as described in 4.0 but without the addition of the Diphenylcarbazide solution. Measure the extinction of this solution in the same way and register it as E_2.

A-7.5 Blank Solution

Fill three quarters of a 25 ml volumetric flask with the extract solution obtained in **A-7.3.1**, add 1 ml of diphenylcarbazide solution (*see* **A-7.2.1**) and 1 ml of phosphoric acid (*see* **A-7.2.3**) and make up to the mark and mix well. Store this solution in a dark and cool place.

A-7.6 Calibration

Prepare calibrating solutions from the standard solution. The chromium concentration in these solutions should cover the expected range of measurements.

A-7.6.1 Derive a suitable calibration curve by using 0.5/1.0/3.0/5.0/8.0 ml of the standard solution prepared as per **A-7.2.7**. Pipette each of these volumes of standard solutions (*see* **A-7.2.7**) separately into 25 ml volumetric flasks. Fill three fourth of the volume of flask with buffer solution (*see* **A-7.2.5**). Following this, add 1 ml diphenylcarbazide solution (*see* **A-7.2.1**) and 1 ml of phosphoric acid (*see* **A-7.2.3**) to each flask. Make up to volume with distilled water. Mix well and allow to stand for 15 ± 5 min. Measure the extinction of the solutions in a 2 cm cell at 540 nm against the blank obtained in **A-7.5**.

A-7.6.2 Plot a calibration graph with hexavalent chromium concentrations in µg/25 ml against the extinctions measured. The hexavalent chromium concentrations represent the *x*-axis, while their extinctions the *y*-axis.

A-7.7 Calculation and Expression of Results

A-7.7.1 Calculation of hexavalent chromium content, C (reported as hexavalent chromium in mg/kg) on-moisture-free basis:

$$C = \frac{\begin{array}{c}\text{conc. of hexavalent chromium}\\ \text{(as read from the plot)} \times 10 \times 100\end{array}}{\text{Weight of sample} \times (100 - M)}$$

NOTE — To find the concentration of hexavalent chromium from the plot, in the case of experimental solution find out the difference of extinctions of E_1 and E_2 of sample (that is $E_1 - E_2$) and use only that to refer the concentration from the plot drawn.

where

C = hexavalent chromium concentration in mg/kg of basic chromium sulphate,

E_1 = extinction of sample solution with diphenylcarbazide,

E_2 = extinction of sample solution without diphenylcarbazide, and

M = moisture content of the sample.

A-8 DETERMINATION OF PENTACHLORO PHENOL (PCP)

A-8.1 Procedure

Transfer about 5 g of the basic chromium sulphate solution into a separating funnel of about 250 ml capacity along with 100 ml water. Add 15-25 ml of sulphuric acid 10 percent *w/v* aqueous solution and ensure that the *p*H of the contents is acidic. Then carry out liquid — liquid extraction using hexane, analytical grade, as the solvent. At first extract with about 25 ml hexane by shaking the contents thoroughly for about 15 min. Then set it aside for clear separation of the layers (*see* Note). Transfer the aqueous layer into another separating funnel and carryout three more extractions of the aqueous layer with hexane of 25 ml each time in the same manner explained earlier.

NOTE — In case of any emulsion being noticed, break the same with the addition of 20-30 ml saturated sodium chloride aqueous solution or follow the steps explained in **4.3.4.2** of IS 14575.

A-8.2 Combine the hexane extracts and dry it by passing through a bed of anhydrous sodium sulphate of about 10 g. The dried hexane layer is evaporated to near dryness by a rotary evaporator.

A-8.3 The residue is taken for acetylation. Then follow the procedure as detailed in Method A of IS 14575.

ANNEX B

(*Clause* 6)

SCALE OF SAMPLING AND CRITERIA FOR CONFORMITY

B-1 SCALE OF SAMPLING

B-1.1 Lot

In any consignment all the containers of the same size of basic chromium sulphate manufactured in a single batch shall constitute a lot.

B-1.2 For ascertaining the conformity of the lot to the requirements of this standard, each lot shall be considered separately. The number of containers to be selected from the lot for this purpose shall depend on the size of the lot and shall be in accordance with Table 2, col 2 and col 3.

Table 2 Scale of Sampling

Sl No. (1)	No. of Containers in the Lot (2)	No. of Containers to be Selected (3)
i)	Up to 25	3
ii)	26-50	4
iii)	51-100	5
iv)	101-200	6
v)	201 and above	8

B-1.3 The containers required to be selected in **B-1.2** from the lot shall be chosen at random from the lot. In order to ensure the randomness of selection, random number tables shall be used (*see* IS 4905).

B-2 PREPARATION OF TEST SAMPLES

B-2.1 From each of the containers selected according to **B-1.3**, a small representative portion of material shall be drawn with the help of a suitable sampling instrument. The material from each container shall not be less than 300 g.

B-2.2 From the material drawn in **B-2.1** from each selected container, equal quantities of material shall be taken and mixed thoroughly to form a composite sample. The composite sample made from small portions of material from all the selected containers shall weigh at least 300 g. It shall be divided into three equal parts of at least 100 g each, one for the purchaser, one for the supplier and the third for the referee purposes.

B-2.3 Remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample representing the container. One set of such individual samples representing the selected containers shall be marked for the purchaser, one for the supplier and third for the referee purposes.

B-2.4 All the individual samples and the composite samples shall be transferred to separate sample containers. The sample containers shall be labelled with full identification particulars.

B-3 NUMBER OF TESTS

B-3.1 Tests for the determination of percentage basicity and chromium content shall be performed on each of the individual samples.

B-3.2 Tests for the determination of all the other characteristics shall be performed on a composite sample.

B-4 CRITERIA FOR CONFORMITY

B-4.1 Individual Samples

B-4.1.1 In respect of the requirements tested on individual samples, the mean ($\overline{X}$) and the range (R) of the test results shall be computed. The mean X shall be the sum of the test results divided by the number of the test results. The range R shall be the difference between the maximum and the minimum of the test results.

B-4.1.2 *Percent Basicity*

From the test results of this requirement ($\overline{X} + 0.4\,R$) and ($\overline{X} - 0.4\,R$) shall be calculated. The lot shall be considered as conforming to this requirement if the values of these two expressions include entirely the specified range.

B-4.1.3 *Chromium Percentage*

From the test results for this requirement the value of the expression ($\overline{X} - 0.4\,R$) shall be calculated. The lot shall be considered to be in conformity for this requirement if the value of the expression ($\overline{X} - 0.4\,R$) calculated above does not fall short of the specified minimum value.

B-4.2 For Composite Samples

In respect of the requirements of this standard tested on a composite sample, the lot shall be considered to be in conformity if all the test results on these characteristics satisfy the relevant requirements specified in this standard.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

Organization	*Representatives(s)*
Central Leather Research Institute, Chennai	Director (***Chairman***)
A. V. Thomas Leather & Allied Products Pvt Ltd, Chennai	Shri Habib Hussain Shir K. Manivannan (*Alternate*)
All India Skins & Hide Tanners and Merchants Assciation, Chennai	Shri Mohan M. Sreenivas Shri S. Mohammed Hassan (*Alternate*)
BASF India Ltd, Mumbai	Shri P. R. Chaudhari Dr S. A. Nadgouda (*Alternate*)
Bata India Ltd, Hathidah Bihar	Dr Sudhir Kumar Das
Central Footwear Training Institute, Agra	Shri S. N. Ganguly Shri S. Chakraborty (*Alternate*)
Central Leather Research Institute, Chennai	Dr C. Muralidharan
Central Pollution Control Board, Delhi	Shri T. Venugopal Shri Ajay Aggarwal (*Alternate*)
College of Leather Technology, Kolkata	Dr Buddhadev Chattopadhyay Prof Swapan Kumar Basu (*Alternate*)
Consumer Federation of India, New Delhi	Representative
Council for Leather Exports, Chennai	Shri M. M. Hashim Dr Zackria Sait (*Alternate*)
Directorate General of Quality Assurance (DGQA), Kanpur	Shri G. C. Kannaujia Shri S. Chakraborty (*Alternate*)
Footwear Design & Development Institute, Noida	Shri B. S. Katiyar Shri Navendu Shekhar (*Alternate*)
Harman Sales Pvt Ltd, Mumbai	Shri H. K. Razdan Shri Jeevan Razdan (*Alternate*)
Indian Finished Leather Manufacturers & Exporters Association, Chennai	Shri Shafeeque Ahmed Shri V. P. Naimmur Rahman (*Alternate* I) Shri M. Salahuddin Bari (*Alternate* II)
Indian Footwear Component Manufacturers Association, Kolkata	Shri Mani Almal
Indian Leather Products Association, Kolkata	Representative
Indian Leather Technologists Association, Kolkata	Shri Arnab Jha Dr Gautam Mukherjee (*Alternate*)
Indian Shoe Federation, Chennai	Shri Ramesh Subramaniam Shri Abhijit Seth (*Alternate*)
Indofil Chemicals Co Ltd, Mumbai	Shri S. K. Jha
International Institute of Saddlery Technology and Export Management, Kanpur	Representative
Italab Private Ltd, Chennai	Shri M. A. Etiappan Shri J. J. Patel (*Alternate*)
Khadi & Village Industries Commission, Mumbai	Shri S. Vijaya Kumar Shri R. Lawrence (*Alternate*)
Kings International Ltd, Kanpur	Shri Taj Alam Shri G. S. Kumaran (*Alternate*)
Leather Chemicals Manufacturers Association, Mumbai	Shri Vaduvur T. Srikanth Shri Sanjeev Mehta (*Alternate*)
Liberty Footwear, Karnal	Shri Adesh Gupta Shri S. S. Lahiri (*Alternate*)
Ministry of Commerce, New Delhi	Representative

Organization	*Representives(s)*
National Institute of Design, Ahmedabad	Shri Shekhar Chatterjee
National Institute of Fashion Technology, New Delhi	Shri E. Sivasakthi
Office of the Development Commissioner, MSME, New Delhi	Shri S. K. Basu Shri R. K. Kapoor (*Alternate*)
Planning Commission, New Delhi	Dr S. C. Lahiri
Robinson Sports, New Delhi	Shri S. S. Jolly
SGS India Pvt Ltd, Gurgaon	Shri P. Venkatesan Ms Monika Sharma (*Alternate*)
Sports Authority of India Ltd, New Delhi	Representative
TALTEIA, Chennai	Shri Vadivelu
Tata International Limited, Dewas	Shri O. K. Kaul Shri R. G. Bhirud (*Alternate*)
The Leather Forum Calcutta for Research and Development, Kolkata	Shri Sanjoy Dasgupta Shri Prasanto Bhattacharjee (*Alternate*)
Vishnu Chemicals, Chennai	Dr P. G. Pradhan Shri C. P. C. Kamalakar Rao (*Alternate*)
BIS Directorate General	Scientist 'F' and Head (CHD) [Representing Director General (*Ex-officio*)]

Member Secretary
Shri E. Devendar
Scientist 'F' and Head (CHD), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc No.: CHD 17 (1618).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.org.in

Regional Offices:		*Telephones*
Central	Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	2323 7617 2323 3841
Eastern	1/14 C.I.T. Scheme VII M, V. I. P. Road, Kankurgachi KOLKATA 700054	2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	SCO 335-336, Sector 34-A, CHANDIGARH 160022	260 3843 260 9285
Southern	C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442 2254 2519, 2254 2315
Western	Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	2832 9295, 2832 7858 2832 7891, 2832 7892

Branches: AHMEDABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM. VISAKHAPATNAM.

Published by BIS, New Delhi